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METHOD OF OBTAINING POLYBENZIMIDAZOPYRROLONES(U)  
FOREIGN TECHNOLOGY DIV WRIGHT-PATTERSON AFB OH  
V V KORSHAK ET AL. 09 AUG 84 FTD-ID(RS)T-0796-84

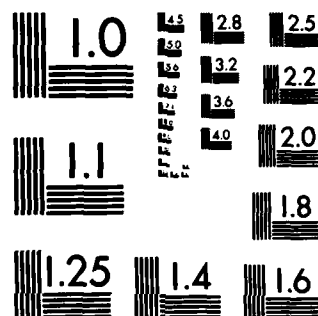
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MICROCOPY RESOLUTION TEST CHART  
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## FOREIGN TECHNOLOGY DIVISION

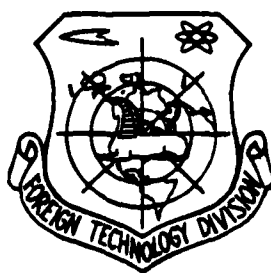
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METHOD OF OBTAINING POLYBENZIMIDAZOPYRROLONES

by

V. V. Korshak, A. L. Rusanov, and R. D. Katsarava



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# EDITED TRANSLATION

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METHOD OF OBTAINING POLYBENZIMIDAZOPYRROLONES

By: V. V. Korshak, A. L. Rusanov, and R. D. Katsarava

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# U. S. BOARD ON GEOGRAPHIC NAMES transliteration SYSTEM

Block	Italic	Transliteration	Block	Italic	Transliteration
А а	<i>А а</i>	A, a	Р р	<i>Р р</i>	R, r
Б б	<i>Б б</i>	B, b	С с	<i>С с</i>	S, s
В в	<i>В в</i>	V, v	Т т	<i>Т т</i>	T, t
Г г	<i>Г г</i>	G, g	У у	<i>У у</i>	U, u
Д д	<i>Д д</i>	D, d	Ф ф	<i>Ф ф</i>	F, f
Е е	<i>Е е</i>	Ye, ye; E, e*	Х х	<i>Х х</i>	Kh, kh
Ж ж	<i>Ж ж</i>	Zh, zh	Ц ц	<i>Ц ц</i>	Ts, ts
З з	<i>З з</i>	Z, z	Ч ч	<i>Ч ч</i>	Ch, ch
И и	<i>И и</i>	I, i	Ш ш	<i>Ш ш</i>	Sh, sh
Й й	<i>Й й</i>	Y, y	Щ щ	<i>Щ щ</i>	Shch, shch
К к	<i>К к</i>	K, k	Ъ ъ	<i>Ъ ъ</i>	"
Л л	<i>Л л</i>	L, l	Ы ы	<i>Ы ы</i>	Y, y
М м	<i>М м</i>	M, m	Ь ь	<i>Ь ь</i>	'
Н н	<i>Н н</i>	N, n	Э э	<i>Э э</i>	E, e
О о	<i>О о</i>	O, o	Ю ю	<i>Ю ю</i>	Yu, yu
П п	<i>П п</i>	P, p	Я я	<i>Я я</i>	Ya, ya

\*ye initially, after vowels, and after Ъ, Ь; e elsewhere.  
When written as ё in Russian, transliterate as yě or ě.

## RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	sinh <sup>-1</sup>
cos	cos	ch	cosh	arc ch	cosh <sup>-1</sup>
tg	tan	th	tanh	arc th	tanh <sup>-1</sup>
ctg	cot	cth	coth	arc cth	coth <sup>-1</sup>
sec	sec	sch	sech	arc sch	sech <sup>-1</sup>
cosec	csc	csch	csch	arc csch	csch <sup>-1</sup>

Russian English

rot curl  
lg log

## GRAPHICS DISCLAIMER

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## METHOD OF OBTAINING POLYBENZIMIDAZOPYRROLONES

Author's certificate number 339558

Authors of invention: V. V. Korshak, A. L. Rusanov, and  
R. D. Katsarava

Applicant: Institute of Hetero-organic Compounds, AS USSR

A method is known for obtaining polybenzimidazopyrrolones by means of the interaction of dianhydrides of tetracarboxylic acids and tetraamines in a solution with subsequent processing of the resulting polyaminoamido acids with acetic anhydride; with further dehydration the number of cross-links of the chains is increased. Using this principle the attempts to increase the solubility of the end products by carrying out the partial hydrolysis of pyrrone, obtained from the dianhydrides and tetraamines in polyphosphoric acid, were unsuccessful.

A method is proposed for obtaining soluble film-forming pyrrones from polyaminoamido acids (PAAK), synthesized in a medium of dimethylformamide (DMFA) at 20°C, cyclized to polyaminoamides under soft conditions in a medium of acetic anhydride at 140-150°C in the presence of pyridine, which then are converted into intermediate pyrrone in a solution of polyphosphoric acid (PFK). Further it is hydrolyzed with mixing of the solution with ice. Polycarboxybenzimidazole is obtained which is free from undesirable cross-links, which then at 140°C in a medium of acetic anhydride is converted into final polybenzimidazopyrrolone, which is readily soluble in sulfuric acid, a mixture of tetrachloroethane and phenol (3:1), polyphosphoric and formic acid.

The polymers obtained have  $\eta_{sp}$  of 0.5% solutions in a mixture of tetrachloroethane and phenol of 0.28-0.54 dl/g. By casting from a solution strong films are obtained.

Example. In a four-neck flask, equipped with a mixer, and inlet and outlet for argon, and a dropping funnel, 4.6055 g (0.02 moles) of 3,3',4,4'-tetraaminodiphenyloxide are dissolved in 28 ml of dry deoxygenated dimethylformamide. To the solution of tetraamine with rapid mixing [they] add a solution of 6.2045 g (0.02 moles) of dianhydride of 3,3',4,4'-diphenyloxidetetra-carboxylic acid in 80 ml of dimethylformamide, whereupon 75-80% of the entire amount is added in the course of 10-15 min, and the remaining 20-25% is poured in by drops in the course of an hour. After addition of the entire amount of dianhydride it is mixed yet for 4-5 hours. The result is a viscous solution of high-molecular polyaminoamido acid ( $\eta_{sp}$  of an 0.5% solution in DMFA at 25°C is 2.6 dl/g.).

To the reaction solution of PAAK in DMFA 32 ml of pyridine and 40 ml of acetic anhydride are added. The solution is heated at a temperature of 150°C for 2 hours. After cooling to room temperature the solution is filtered and the polymer precipitated with water. The polymer which has precipitated in the form of fiber is filtered, washed thoroughly with water, then with acetone and dried in a vacuum-drier. The result is high-molecular polyamidoimide ( $\eta_{sp}$  of an 0.5% solution in dimethylformamide is 1.76 dl/g.).

The 2 g of polyamidoimide is filled with 20 g of 116% PKF and slowly heated to 200°C. When 80-100°C is reached the mixer is turned on. A dark-red solution of polymer in polyphosphoric acid is formed. When 200°C is reached the solution begins to darken and by the end of the reaction (after 2 hours at 200°C) has a dark color. The hot reaction solution is decanted in water with ice. The polymer which precipitated in the form of small granules of a yellowish color is filtered and washed with water up to a neutral reaction, and then with acetone and dried in a vacuum at room temperature. The result is polycarboxybenzimidazole.

The product obtained is poured over 50 ml of acetic anhydride and boiled for 8-10 hours. After cooling to room temperature, the polymer of a yellowish-tobacco color which is formed is filtered, washed with acetone and dried in a vacuum at room temperature.

The polymer obtained has  $\eta_{sp}$  of an 0.5% solution in a mixture of tetrachloroethane and phenol at 25°C of 0.54 dl/g, and in H<sub>2</sub>SO<sub>4</sub> - 0.42 dl/g.

From the solution in a mixture of tetrachloroethane and phenol by the method of casting on glass backings, transparent golden-yellow, elastic strong films are obtained (elongation 7-10%, tensile strength 850 kg/cm<sup>2</sup>).

#### Subject of invention

A method of obtaining polybenzimidazopyrrolones by means of the interaction of dianhydrides of tetracarboxylic acids with tetraamines in a solution with subsequent treatment of the polyaminoamido acid obtained with acetic anhydride with heating, characterized by the fact that for the purpose of obtaining a soluble linear polymer the product obtained is successively heated in polyphosphoric acid at 200°C, subjected to hydrolysis and treated with acetic anhydride with heating.

**END**

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